Indian Head Division
Naval Surface Warfare Center
Indian Head, MD 20640-5035

IHTR 2310
15 January 2001

DISCUSSION OF PROCESS EFFECTS ON EX-99 PROPELLANT CHARACTERISTICS AND CONTINUOUS EXTRUSION BEHAVIOR

F.M. Gallant

REPORT DOCUMENTATION PAGE

1. REPORT DATE (DD-MM-YYYY) 15-01-2001	**2. REPORT TYPE** Final Report	**3. DATES COVERED (FROM - TO)** xx-xx-2001 to xx-xx-2001
4. TITLE AND SUBTITLE DISCUSSION OF PROCESS EFFECTS ON EX-99 PROPELLANT CHARACTERISTICS AND CONTINUOUS EXTRUSION BEHAVIOR Unclassified		**5a. CONTRACT NUMBER** **5b. GRANT NUMBER** **5c. PROGRAM ELEMENT NUMBER**
6. AUTHOR(S) Gallant, F. M. ;		**5d. PROJECT NUMBER** **5e. TASK NUMBER** **5f. WORK UNIT NUMBER**
7. PERFORMING ORGANIZATION NAME AND ADDRESS Indian Head Division Naval Surface Warfare Center Indian Head , MD 20640-5035		**8. PERFORMING ORGANIZATION REPORT NUMBER** IHTR 2310
9. SPONSORING/MONITORING AGENCY NAME AND ADDRESS ,		**10. SPONSOR/MONITOR'S ACRONYM(S)** **11. SPONSOR/MONITOR'S REPORT NUMBER(S)**
12. DISTRIBUTION/AVAILABILITY STATEMENT A PUBLIC RELEASE ,		

13. SUPPLEMENTARY NOTES

14. ABSTRACT

This report documents the process study for the production of EX-99, in a twin-screw extruder. Using powder supplied by Bofors AB, this was the first effort in the United States to produce this formulation in a continuous process. For reasons of quality and efficiency, a factorial experiment strategy was employed to quantify the effects of screw design, solvent content, extruder speed, and process temperature. This study was unique in that samples from each of the 12 treatment conditions were submitted for test and evaluation. Using regression analysis, a number of predictive models were presented for characteristics such as, dry grain diameter, perforation diameters, web thickness, occurrence of white patches, die pressure, and extruder torque. The importance of the experimental approach is illustrated not by the knowledge of which factors are important to which measurements, but by the power to predict results at untested process conditions. Two parameters, inner and outer web diameter, were jointly solved to find the process conditions that minimized the difference between the two dimensions. Incorporating the lead engineer?s input as a basis for process conditions, an optimal setting was calculated. This new setting was the basis for a 600-pound production run with good results.

15. SUBJECT TERMS

Twin-screw extruderSPCEX-99Continuous processingDesign of experimentsHelovagun propellantscreening experiment

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
			Same as Report (SAR)	26	Simpson, Susan simpsonsm@ih.navy.mil
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER International Area Code Area Code Telephone Number 301 744-4284 DSN 354-

REPORT DOCUMENTATION PAGE

Form Approved
QMB No. 0704-0188

1. AGENCY USE ONLY (*Leave Blank*)	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	15 January 2001	Final Report

4. TITLE AND SUBTITLE

DISCUSSION OF PROCESS EFFECTS ON EX-99 PROPELLANT CHARACTERISTICS AND CONTINUOUS EXTRUSION BEHAVIOR

5. FUNDING NUMBERS

6. AUTHOR(S)

F.M. Gallant

7. PERFORMING ORGANIZATIONS NAME(S) AND ADDRESS(ES)

Indian Head Division
Naval Surface Warfare Center
Indian Head, MD 20640-5035

8. PERFORMING ORGANIZATION REPORT NUMBER

IHTR 2310

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; distribution is unlimited.

12b. DISTRIBUTION CODE

13. ABSTRACT (*Maximum 200 words*)

This report documents the process study for the production of EX-99, in a twin-screw extruder. Using powder supplied by Bofors AB, this was the first effort in the United States to produce this formulation in a continuous process. For reasons of quality and efficiency, a factorial experiment strategy was employed to quantify the effects of screw design, solvent content, extruder speed, and process temperature. This study was unique in that samples from each of the 12 treatment conditions were submitted for test and evaluation. Using regression analysis, a number of predictive models were presented for characteristics such as, dry grain diameter, perforation diameters, web thickness, occurrence of white patches, die pressure, and extruder torque. The importance of the experimental approach is illustrated not by the knowledge of which factors are important to which measurements, but by the power to predict results at untested process conditions. Two parameters, inner and outer web diameter, were jointly solved to find the process conditions that minimized the difference between the two dimensions. Incorporating the lead engineer's input as a basis for process conditions, an optimal setting was calculated. This new setting was the basis for a 600-pound production run with good results.

14. SUBJECT TERMS

Twin-screw extruder	SPC	EX-99	Continuous processing
Design of experiments	Helova	gun propellant	screening experiment

15. NUMBER OF PAGES

26

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	SAR

This page intentionally left blank.

FOREWORD

The work reported herein was performed at the Indian Head Division, Naval Surface Warfare Center, Indian Head, MD.

R.R. Nuckols
Manager, Environmental and
Process Technology Branch

Approved and released by:

C.F. Clark
Director, Explosives and Propellant Division

This page intentionally left blank.

CONTENTS

Tables

Figures

This page intentionally left blank.

INTRODUCTION

Regardless of the product, the manufacture of any material using a complex process requires understanding the effects of process conditions upon the quality of the product. In the specific case of processing EX-99 powder into grains of gun propellant using a continuous twin-screw extrusion process, there was no knowledge regarding the effect of conditions upon ballistics or characteristics. If there are effects, what is the magnitude and how does one achieve optimum performance? Judicious application of design of experiments techniques can quantitatively address these questions, guide product optimization, and control quality of manufacturing.[1] This report documents the analysis of an investigation into the effects of extruder processing conditions on the mixing, extrusion, physical characteristics, and ballistics of EX-99 gun propellant.

The source of the powder is Bofors Explosives. Last year a small lot was received to summarily evaluate the extrusion process in the 40-mm twin-screw extruder at Indian Head. During the May 1999 trials, 5 pounds of grains, produced at a rate of 30 lb/hr with 11 percent by weight solvent,* were subjected to the standard battery of tests.[2] Some of the test results did not meet the draft specification and could not be readily explained. Were the results a function of the process or the powder?

In the last decade, Naval Surface Warfare Center, Indian Head Division has gained a wealth of knowledge and experience in the twin-screw processing of solvent-based gun propellants, namely, XM-39 and XM-43.[3] These studies have concentrated mostly on the effect of processing conditions upon process responses; e.g., die pressure, viscous heating, rheology, and work energy. While ballistics and other characterization data were collected for the extruded samples, there were no studies to quantifiably correlate the test results to intentional process changes. Instead, the approach was to produce a variety of samples at various process conditions according to designed experiments and characterize only the few winners of the beauty contest. The result of that approach was only to provide an indication of a twin-screw capability for producing high quality grains. What was neglected was a sensitivity determination of the physical and ballistic properties to the process.

There is no question that continuous processing using a twin-screw extruder is a complex process. There are numerous combinations of process configuration, temperature profiles, screw designs, feeding options, solvent ratios, ingredient preparation, etc. The power of designed experiment techniques is that any number of process factors or conditions can be statistically evaluated in the most cost- and time-efficient manner. The typical approach is to first conduct a screening experiment to identify the few key factors from all the possibilities. Next, an optimization study can be designed using only the most important factors. Importantly, these follow-on studies can quantify any interactions between the factors. The subject of this report is the analysis of the data from the EX-99 screening experiment.

*All mention of solvent concentration in this report is understood to be by weight.

DATA ANALYSIS METHOD

There is more than one method of analyzing data from a screening experiment. The selection depends upon the data quality, quantity, desired resolution of results, and the experimental design. Several methods of analysis were employed for this study. The most widely used method is analysis of variance[4] (ANOVA), which will be explained by example. Other types of analysis include Bayesian methods.[5] Graphical methods are usually employed to suggest trends or effects not rigorously supported by the model.[6]

The overall benefit of analysis is to answer two questions. First, is the factor significant and to what degree? For example, does the solvent concentration affect the die pressure, and how strong is that effect? How does the effect of solvent compare to the effect of temperature on die pressure? The second question answered is the direction of that effect. For example, if high die pressure results in better density, then what settings of solvent and die temperature optimize this effect? Does pursuing this optimization compromise the safety of the operation? An experimental approach based on the designed experiments techniques yields quantifiable answers to these types of questions. The purpose of this report is to summarize the results of the EX-99 extrusion screening study in a manner consistent with scientific scrutiny and rigorous statistical treatment. Because a screening design was employed, only the main effects can be estimated with accuracy.

Screening Design

Previous designed experiments conducted with XM-43 were also screening designs; however, the analyses focused only on process responses. Because of budget restraints, a complete set of samples was never previously tested. Without any quantified data for ballistics or homogeneity, we have no knowledge base to relate physical characteristics of the propellant back to the process. Therefore a decision was made to start building that knowledge base with this study.

A screening design was chosen because it represents the best cost trade-off between minimizing the number of samples to test and maximizing the number of factors to screen for any positive or negative effects. The final four factors chosen were solvent content, extruder screw speed, temperature, and screw geometry. Throughput was originally considered as well, but was dropped from the design for unknown reasons. The solvent content, screw speed, and temperature were evaluated at two levels—i.e., one high and one low. Each of these was crossed with three screw designs resulting in the matrix shown in Table I, which guided the extrusion schedule.

Table I. Screening Design Matrix

Identification	Solvent concentration (%)	Extruded die temperature (°F)	Extruder speed
Screw Geometry-Screw Design 45			
A	11.5	90	80
B	11.5	120	110
C	12.5	120	80
D	12.5	90	110
Screw Geometry-Screw Design 46			
E	11.5	120	80
F	11.5	90	110
G	12.5	90	80
H	12.5	120	110
Screw Geometry-Screw Design 48			
I	12.5	110	90
J	11.5	80	90
K	12.5	80	120
L	11.5	110	120

Analysis: Physical Effects

In the analysis of experimental data, an investigator desires to interpret the results in terms of the controls. This is a method of modeling. For example, an experimenter may want to explain die pressure in terms of the process controls. Assuming linear relationships, a model is constructed that is the sum of weight terms multiplied by the factors plus a value for the error.

In this report the models are given in two forms. The regression form eases interpretation but is cumbersome for calculating the predicted effect. The intercept represents the average for all conditions. The weights and their signs are relative in the regression model. For example if weight *a* is twice weight *b*, then factor *A* contributes to the estimate twice as much as factor *B.* If the sign of weight *a* is positive, then the estimate will increase as factor *A* is changed from low to high and vice versa. The second form is a simplification of the regression model that allows easier calculation of the estimate but no interpretation from the terms. (In some instances the screw geometry factors were negligible and thus were included in the intercept terms of the regression model at the author's discretion. If the regression models that follow do not include the screw geometry, then assume the terms were included in the intercept.)

Note that while all the weights are presented for each regression model, not all are statistically significant. The significant, or power, factors are identified for each. The insignificant factors are still part of the estimate—just a statistically insignificant part.

Responses, or predictions, can be estimated with good precision as long as the model is valid. In this context, the validity of a model is expressed as the probability that it does not provide a statistically valid estimate. One convention is that for probabilities greater than 5 percent, the model is judged inadequate. A value of 5 is conservative and chosen somewhat arbitrarily. In any case it is safe to assume that the lower the probability for rejection, the better the estimates from the model.

The data from the ballistics testing and physical characterization were analyzed to determine if the property could be explained in terms of the experimental design. The properties that provided good estimates are discussed in detail. Those that had probability values greater than 5 percent, but only slightly greater, were classified as fair models. In other words, they are only fair at estimating the effects. Good models were found for the average dry diameter, outer/inner perforation diameters, inner web thickness, and the number of white patches. At best, only a fair model could explain the outer web thickness.

Good Models

Diameter (Dry) : The average dry diameter can be controlled by the process parameters. While screw geometry had no effect, temperature and solvent had significant, equal, negative effects. The extruder speed had a significant but lesser effect. Note the magnitudes and signs of the respective terms in the regression model below. The probability that the model does not adequately describe the data is very low at 0.08 percent.

$$Diameter = 0.404000 - 0.003333\frac{solvent - 12}{0.5} - 0.003500\frac{temperature - 105}{15} + 0.001500\frac{rpm - 95}{15}$$

In this example, the average diameter for all conditions was 0.4040 inch. Since the coefficients for solvent and temperature are negative, an increase in either (or both) should cause a decrease in the grain diameter. The magnitude of the effects are roughly equal (−0.0033 versus −0.0035). The factor, rpm, has a positive influence on the grain diameter, although from its lower coefficient (0.0015) it has approximately half the influence of solvent or temperature.

For predicting the dry diameter, this simplifies to the following for $screw_{48}$:

$$Diameter = 0.498992 - 0.006666 solvent - 0.0002333 temperature + 0.000100 rpm$$

For example, percent solvent, temperature, and rpm at levels of 11.5, 90, and 80 respectively predict an average dry diameter of 0.409 inch. The experimental average value for these conditions was 0.408 inch.

Average Diameter of Outer Perforation: In consideration of perforation size as a group, there was a difference in the averages between the outer perforation (perf) and center perf. Reasonable models were found for both measurements. Screw geometry was identified as contributing to both as well. However, one must recognize the possibility that the effects attributed to screw geometry are confounded with any day effects. The result is this caveat. If all three screws are found to be statistically significant, then that significance can be equally attributed to any day-to-day variation. Why? Most of the test conditions using the same screw configuration were conducted on the same day. The way to resolve the ambiguity is to replicate the extrusion trials on different days and model the day effect as a separate influence. However, to achieve full resolution increases experiment costs and reduces facility efficiency. It is conventional to assume that the confounded effects are small and much less significant than the design factors.

The model for the average perf diameter of the outer perforations indicated that temperature and rpm had effects as strong as the screw geometry. The probability that the parameters are not statistically valid to interpret the data is 0.25 percent. There was no apparent interaction between temperature and rpm. The model for the outer perf diameter is given by the following.*

$$OuterPerf = 0.018833 - 0.000783(screw_{46} - screw_{45}) + 0.001092(screw_{48} - screw_{45}) - 0.000167\frac{solvent - 12}{0.5}$$
$$-0.000850\frac{temperature - 105}{15} + 0.000717\frac{rpm - 95}{15}$$

This equation simplifies to the following for the case of $screw_{48}$.

$$OuterPerf_{screw48} = 0.025342 - 0.0003335 solvent - 0.0000567 temperature + 0.0000478 rpm$$

Average Diameter Center Perf: Note the discussion above with regard to the observations of the outer perf diameter. It is interesting that the size of the center perforation seems to be a function of the screw geometry and, to a much lesser extent, solvent. (Recall that screw geometry was confounded with day-to-day variation if any.) Temperature and rpm were not significant factors. The model is significant with a probability of rejection at 0.13 percent and is represented by the following.

$$CenterPerf = 0.016533 - 0.000633(screw_{46} - screw_{45}) + 0.001267(screw_{48} - screw_{45}) - 0.000183\frac{solvent - 12}{0.5}$$
$$-0.000033\frac{temperature - 105}{15} + 0.000083\frac{rpm - 95}{15}$$

Fixing the screw geometry on $screw_{48}$, the simplified equation follows.

$$CenterPerf_{screw48} = 0.0229527 - 0.000366 solvent - 0.00000222 temperature - 0.00000556 rpm$$

Inner Web Thickness: The data indicate that there is a good model for the inner web diameter. The probability that the model does not describe the data is 0.09 percent. The significant effects were solvent, temperature, and rpm in that order. Note there are no interactions between neither solvent concentration and barrel temperature nor between solvent concentration and extruder rpm. The model is given as follows.

$$I_1 = 0.09088 - 0.000383(screw_{48}) - 0.002267\frac{solvent - 12}{0.5} - 0.002083\frac{temperature - 105}{15} + 0.001667\frac{rpm - 95}{15}$$

Since the screw design was later fixed on geometry Number 48 for the production runs, the value for $screw_{48}$ was set equal to one resulting in the following predictive equation:

$$I_{screw48} = 0.148928 - 0.004534 solvent - 0.00013887 temperature + 0.00011113 rpm$$

*To evaluate an equation with screw geometry as a factor, use a value of 1 for the screw to be modeled and 0 for the others.

Fair Models

ln(White Spots): Early in the investigation the occurrence of white patches was observed on most dried samples, though the frequency seemed to vary widely with the process conditions. The patch was identifiable by color only. All samples were subjected to analysis by scanning electron microscope (SEM) and Fourier transform infrared (FTIR) microreflectance spectroscopy, giving particular attention to the white patches among other criteria used for the SEM quality assessment of Helova grains. The results of the SEM study were not quantitative, and there was little physical difference among all samples.[7] The chemical composition of the spots were similar to the surrounding materials except for a higher concentration of cellulose acetate butyrate (binder) present in the white patch.[8]

It was the prevalence of the patches that prompted the incorporation of a third screw design, which was designated as $screw_{48}$, for evaluation. Analysis of the frequency data clearly indicated that screw geometry alone controlled the appearance of the spots. The best model was given by the logarithmic transform of the frequency and had a probability of failure equal to 0.067 or 6.7 percent. The predictive model is given by the following.

$$\ln(spots) = 1.5012 + 0.89408(screw_{46} - screw_{45}) - 1.8521(screw_{48} - screw_{45}) + 0.22734\frac{solvent - 12}{0.5}$$
$$+0.25335\frac{temperature - 105}{15} + 0.31001\frac{rpm - 95}{15}$$

How were the spots quantified? The frequency of occurrence was determined for each condition. This was done by choosing a fixed number of grains at random and counting the total number of discrete white patches visible on the grain exteriors. The individual averages are provided in Table II. Note that the latest screw design effectively reduced the occurrence of white spots.

Table II. Average Number of White Patches per Grain for Each Condition

$Screw_{45}$		$Screw_{46}$		$Screw_{48}$	
Condition	Average	Condition	Average	Condition	Average
A	3.5	E	3.0	I	0.16
B	9.0	F	23	J	0.16
C	18	G	7.0	K	1.6
D	33	H	30	L	1.6

The regression model is simplified by fixing the screw geometry and reducing the remaining terms yielding the following expression.

$$\ln(spots_{screw\,48}) = -9.5439 + 0.45468 solvent + 0.01689 temperature - 0.020667 rpm$$

A plot of the model's prediction versus the observed percentage of white patches is given in Figure 1. The better the prediction, the closer the predicted points are to following a straight line. (This is true for linear models only.) Figure 1 compares the points to an imaginary line and associated 95 percent confidence limits. The plot could be difficult to understand because the transformed data were plotted. Using condition *K* as an example, the white spot count prediction, based on the model for screw geometry and processing conditions, was $e^{-0.1802}$ or 0.84 percent. The actual count for condition *K* was 1.60 percent or $e^{0.4700}$. Likewise condition *H* had a high count of 30 percent, while the model predicted $e^{3.1861}$ or 24 percent.

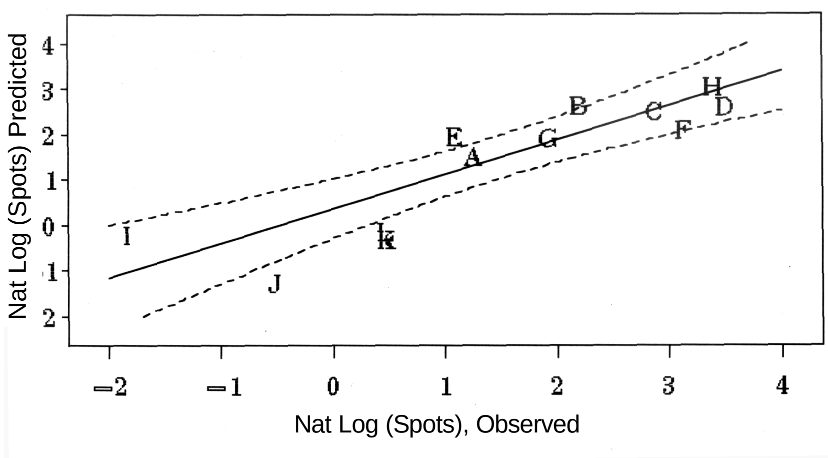

Figure 1. Plot of the Predicted Occurrence of Spots as a Function of the Observed Average Occurrence of Spots for Each Condition

The model is fair at predicting the number of white spots. A more precise method of quantifying the white spots may have resulted in a cleaner model, e.g., a measure of the total area of white patches as a function of the free surface area. Clearly that was unnecessary and certainly would have been wasteful of resources. The results of this analysis show that the conclusion is statistically valid and the means are justified. The method used to quantify the patches adequately determined the cause and provided the lead engineer the direction for quality improvement. Note the dramatic improvement by changing the screw geometry. Conditions I through L (with the more intensive mixing section) are characterized by a lower spot count.

Outer Web Thickness 1: The data indicate that a model can be used to predict the outer web diameter; however, the probability that the model fails is somewhat higher than that for the inner web thickness. The probability for failing is 4.97 percent. The model is still useful judging by the plot of predicted versus observed outer web diameter, see Figure 2.

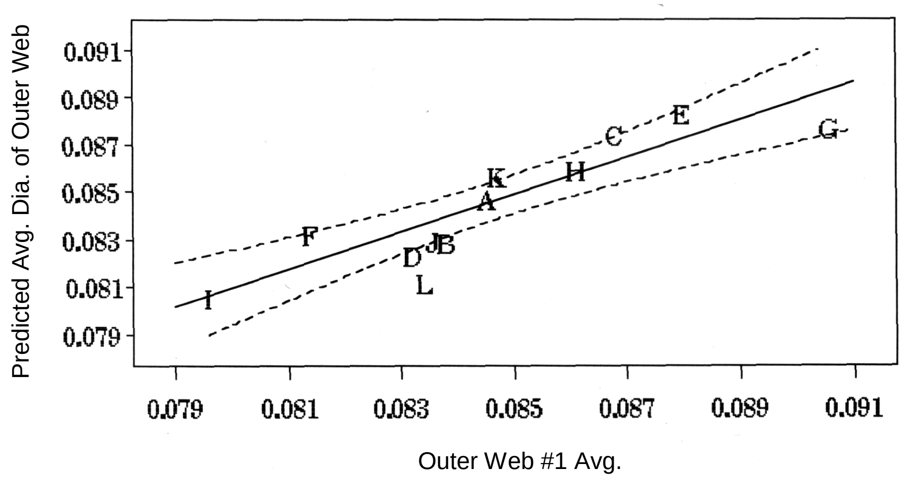

Figure 2. Plot of the Predicted Outer Web Diameter as a Function of the Observed Outer Web Diameter

The model equation is given as follows.

$$OW = 0.084642 + 0.001883(screw_{46} - screw_{45}) - 0.001817(screw_{48} - screw_{45}) + 0.000525\frac{solvent - 12}{0.5}$$
$$+0.000825\frac{temperature - 105}{15} - 0.001725\frac{rpm - 95}{15}$$

Note that the extruder rpm and screw geometry effects are the only statistically significant effects ($p < 0.05$). This simplifies to the following.

$$OW_{screw48} = 0.075375 + 0.00105 solvent + 0.000055 temperature - 0.000115 rpm$$

Poor Models

Although some effects could not be predicted by the factors of this experiment, they bear mention for their uniformity as well as to provide insight for future investigations. Many of these effects are important characteristics with regard to use and performance. The fact that they cannot be described in terms of screw geometry, solvent concentration, process temperature, or extruder rpm implies that they were factors of variables outside this experiment or rigorously unaffected by changes in the process conditions. Poor is not to be interpreted as bad. If there is not a significant range in results, no model is necessary. If the results consist of a reasonable range but result in a poor fitting model, there is another possibility. The response is affected by a variable outside the experiment's controls; i.e., an uncontrolled source of variation. These findings are of interest to those planning similar studies or conducting optimization of this effort.

One example of a response that was unaffected by changes in the process is the dry density of the grains. However the range in average density values was small—from 1.645 to 1.649$\frac{gm}{cm^3}$. One could conclude that the uniformity of the density among the 12 conditions is a testament of its insensitivity to the process design window investigated. Another physical measurement of interest was the average grain length. Since it is an important attribute, there was interest in whether any of the experimental factors had a determinable effect upon the average grain length. The average value for each condition ranged from 0.4050 to 0.4113 inch. There was no statistically valid influence.

The laboratory reports a calculated measure called length uniformity. It is not frequently used, and there was no readily available explanation as to how it is calculated. Although its values ranged from 0.61 to 2.32, there was no correlation with the process conditions. Therefore its precise definition was not pursued.

Analysis Effects: Process Effects

Process effects comprise the material and equipment responses that are measured during the mixing and extrusion. These are important for reasons of safety, quality, and efficiency. The material responses include various material pressures in the mixing sections and the die and associated material temperatures at those same locations. Key parameters are magnitude and stability. The other type of process response is equipment related. The best example is the extruder torque. The torque is a direct indication of the work energy to mix and extrude the material.

Good Models

Die Pressure: Die pressure is one of the most important parameters for monitoring the safety and stability of an extrusion process. In the case of EX-99 and similar propellants, the die pressure is a function of the processing conditions. An excellent model describes the average die pressure. All factors, except for screw geometry, influenced the die pressure according to the following.

$$pdie = 608.6 - 0.6(screw_{46} - screw_{45}) + 9.9(screw_{48} - screw_{45}) - 63.8\frac{solvent - 12}{0.5}$$
$$-88.1\frac{temperature - 105}{15} - 29.6\frac{rpm - 95}{15}$$

Note that all the process effects influence the pressure in the same direction. As each is increased, it has a negative influence upon the die pressure. For example, increasing the extruder rpm from 80 to 110, all other factors remaining the same, decreases the die pressure by approximately 30 psig. Temperature had the greatest effect upon the die pressure. Overall the die pressures for all of the conditions were well-behaved and less than 900 psig.

Because the screw geometry has a negligible effect, fixing the screw geometry on $screw_{48}$ results in the following simplified model.

$$pdie_{screw48} = 2952.7 - 127.5 solvent - 5.873 temperature - 1.973 rpm$$

A plot of the predicted pressures as a function of the experimental (or observed) die pressure illustrates how well this model performs (Figure 3). Note that the high pressure conditions, *F*, *A*, and *J*, each represent the three conditions with the combination of low solvent and low temperature. Likewise *H*, *C*, and *K* represent the opposite. The solid line is an imaginary best fit line, and the dotted lines are the 95 percent confidence limits. The plot of condition *I* is obscured by *E*.

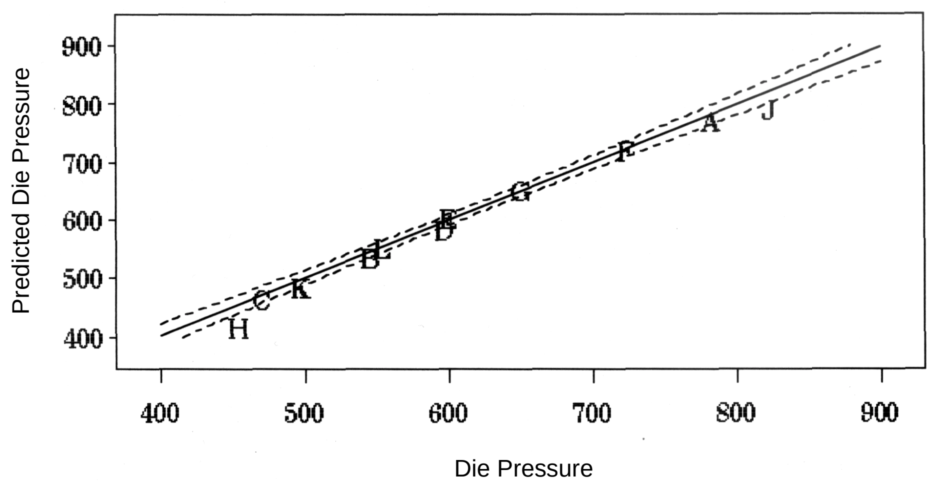

Figure 3. Plot of the Predicted Die Pressure as a Function of the Observed Die Pressure

Extruder Torque: Equally vital to safety as die pressure is the extruder torque. Like pressure, the torque is important for process stability and safety. The model for the torque is interesting in that all process factors make a statistically valid contribution to the prediction. The probability that the model results can be explained by random variation is 0.12 percent. The torque is predicted by this equation.

$$zsk\_torque = 182.083 - 14.08(screw_{46} - screw_{45}) + 25.42(screw_{48} - screw_{45}) - 8.083\frac{solvent - 12}{0.5}$$
$$-14.25\frac{temperature - 105}{15} - 8.917\frac{rpm - 95}{15}$$

This simplifies to the following.

$$zsk\_torque = 532.1 - 14.08(screw_{46} - screw_{45}) + 25.42(screw_{48} - screw_{45}) - 16.166 solvent$$
$$-0.950\,temperature - 0.59447\,rpm$$

In a comparison of geometry alone, $screw_{48}$ contributed to increasing the torque while $screw_{46}$ (the neutral design) was characterized by the lowest values. This is not intuitive in the light that $screw_{48}$ is a blend of the other two designs. One would expect that if $screw_{46}$ has the lowest torque, then $screw_{45}$ would have the highest torque with $screw_{48}$ between the two. However if one takes into account that $screw_{48}$ features the combination of a neutral element followed by a reverse mixing element, then the higher torque can be attributed to this difference.

Poor Models

Material temperatures were measured at locations along the mixing sections and the die. Interestingly, there were no significant temperature changes. As expected, the temperature of the barrel clearly dominates material temperatures. In that EX-99 can be cooled by solvent evaporation and has a relatively low viscosity, it would be unusual to see a high temperature rise in most locations. An error in the experimental setup led to the placement of a probe in the wrong location. Hence there was not a probe in the most likely location to incur a measurable temperature rise if one were to occur. This omission equally applies to pressure data from the most intense mixing section. Hence, no models could be analyzed for temperatures and pressures along the mixing sections.

DIRECTIONS FOR OPTIMIZATION

Web Dimensions

In many respects the measurable characteristics that affect performance, density, and homogeneity were largely unaffected by changes in the process (within the ranges evaluated). A review of the dimensional data prompted a desire to minimize the differences in the thickness of the inner and outer webs. As discussed above, the process configuration and conditions affect the web dimensions. This problem was solved as a linear program that utilized the models for inner (*I*) and outer (*O*) web dimensions by finding the square of the difference and was constrained by the process limits. The results of the minimization agree with observations. The difference between the thickness measurements is eliminated* using the combination screw (Number 48) at 12.25 percent solvent, 120 °F and 80 rpm. These process settings were recommended for the production of 1,000 pounds of grains. At opposing conditions of low temperature and high rpm, the differences were greater than 0.011 inch regardless of solvent content.

The prediction for the best grain geometry points to a corner of the design window for temperature and rpm. It suggests that our design window was constrained in those two dimensions. However, the setting for solvent was not up against a constraint. This is a good result because it demonstrates the ability of the models to find an optimum point within the window. For future optimization experiments of other parameters, the author recommends exploring the space that includes temperatures higher than 120 °F and extruder screw speeds lower than 80 rpm.

For perforation dimensions, the recommended conditions are only the basis for decision making in regard to production of EX-99. Because it was possible to model the effects, a prediction would likely be valid anywhere within the design space. The first question is what impact do these model-suggested conditions have upon safety, quality, and strand handling. Quality and safety have already been demonstrated at the process extremes. Input from the lead engineer indicated that strands with 12.25 percent solvent at a temperature of 120 °F may be too soft for reliable flow through the take-away apparatus. A maximum solvent content of 12 percent was preferred. However the optimization indicates that the difference in web dimensions will increase if the solvent content is lowered.

*Model predicts a difference of less than 0.5 thousandths of an inch.

In situations such as these, the model optimization can pinpoint the best combinations of temperature and rpm with fixed solvent to equalize the inner and outer web dimensions. The model still predicted that the best settings of temperature and speed (with solvent fixed at 12.0 percent) were still at the vertex of the window. However the difference in web dimensions was not optimum. Furthermore there was a concern about a phenomenon locally referred to as resonance pressure flow* that occurs at 80 rpm under some process conditions. By setting the extruder rpm slightly below 80; e.g., 75, the resonance disappears and the difference in web dimension further decreases. The predictions from this series of decisions are presented in Table III.

Table III. Effect of Process Decisions on the Web Dimensions Shown as the Difference Between the Inner and Outer Web Dimensions

Solvent percentage	Process temperature (°F)	Extruder rpm	Inner web –outer web	Comment
12.50	120	80	–0.0014	Condition K
12.25	120	80	0.0000	Optimum within design space
12.00	120	80	0.0014	Take-away considerations
12.00	120	75	0.0002	Recommended optimum
12.00	110	75	0.0022	Production conditions

Upon review of the process data, the author determined that the recommended, optimized conditions were not executed. Instead, the temperature was independently decreased to 110 °F without optimizing the web difference with a new value for the extruder speed. The predicted effect on the web difference is given in Table III. Had the combination of the new temperature and chosen solvent concentration been optimized, the recommended extruder speed would have been 65 rpm. The predicted difference with this set of conditions is 0.0000 inch. The effects of the actual production conditions are discussed in the following section.

*This is the author's term for a type of material behavior in the mixing zone of the extruder. A pressure trace over time of a sensor located above kneading elements will exhibit oscillations of 3 seconds or less. Under certain combinations of screw geometry, rheology, and extruder speed, there will be longer period oscillations in conjunction with the rapid ones on the order of 20 to 80 seconds. Higher periods than 80 seconds are not uncommon. Judicious use of pressure transducers along the barrel easily allows monitoring pressure flow through the extruder. The theoretical basis for these has yet to be fully determined, but there is no negative consequence in the case of EX-99. However, until the phenomenon is better understood, steps are taken to mitigate the occurrence.

RESULTS OF PRODUCTION

In April and May 2000, approximately 700 pounds of EX-99 were produced in two long extruder runs resulting in two lots of propellant. The pertinent data collected to date are summarized in Table IV. Using the rough models based on the screening runs, predictions were made for the extruder process settings.

Table IV. Comparison of Observed Measurements with Predicted Settings

Property	Lot IH940-OOE-EX99-0088	Lot IH940-OOE-EX99-0089	Predicted
	Observed	Observed	
Dry diameter (in)	0.4007	0.4017	0.4008
Outer perf diameter 1 (in)	0.0168	0.0168	0.0187
Center perf diameter (in)	0.0171	0.0167	0.0177
Inner web thickness (in)	0.0875	0.0874	0.0876
Outer web thickness 1 (in)	0.0863	0.0865	0.0854
Die pressure (psig)	605	—	628
Extruder torque (in-lb)	210	—	—

A comparison of the predicted and the observed physical properties shows very good agreement, especially in consideration that the models were generated from a screening study. To handicap matters, the design was augmented with another screw design and four more runs after the initial screening experiment was concluded. Once again it is shown that there is power and flexibility using SPC methods. The predictions indicate that there is value to using the screening data in making process decisions. The best models gave the best estimates of the observed properties with the exception of the outer perf diameter. Even the prediction for the outer web diameter, which was estimated by a poor model, was within several thousandths of an inch.

CONCLUSIONS

This report documents the process study for the production of EX-99 in a twin-screw extruder. This is the first effort in the United States to produce this formulation in a continuous process. For reasons of quality and efficiency, a factorial experiment strategy was employed to quantify the effects of screw design, solvent content, extruder rpm, and process temperature. This study was unique in that samples from each of the twelve treatment conditions were submitted for test and evaluation. In previous studies, only the best few samples were tested with the consequence that the effect of the process on test results could not be quantified or estimated. This study will be a hallmark for future investigations of the continuous processing of Lova gun propellants, not just EX-99. Future studies can begin with response surface methods to develop true models for optimization and control.

Using regression analysis, a number of predictive models were presented for characteristics such as, dry grain diameter, perforation diameters, web thickness, occurrence of white patches, die pressure, and extruder torque. The dry density was uniform across all conditions over the range of tested conditions. Some of the material responses such as material temperature at the die were also uniform over the range of conditions (aside from influence by the process temperature). The cut grain length was independent of the four process factors; this conclusion was anticipated.

The importance of the experimental approach is illustrated not by the knowledge of which factors are important to which measurements, but by the power to predict results at untested process conditions. This power was taken one step further. Two parameters, inner and outer web diameter, were jointly solved to find the process conditions that minimized the difference between the two dimensions. The lead engineer used this optimal setting to trade off undesirable side effects by deviating slightly from the optimal settings. Using the lead engineer's input as a basis, a new optimal setting was predicted that lies slightly outside of the design space. This new setting was the basis for a 600-pound production run with good results.

REFERENCES

1. Beauregard, M. and Schroeder, W., *Application of Design of Experiments to Continuous Processing*, NSWCDD/TR-92/110, Apr 1992, NSWC, Dahlgren, VA.

2. NSWC/Indian Head Division 9420 Memo, Ser 9420A/039/cm, Subj: Monthly Status Report for the CLEVER Project, Indian Head, MD, 14 Jul 1999.

3. Gallant, F.M. et al, *On-Line Rheology* of HELOVA XM-43 Using a Slit-Die Rheometer, IHTR 1805, 11 Aug 1995, NSWC, Indian Head, MD.

4. Hicks. *Fundamental Concepts in the Design of Experiments*, 4th Ed., Oxford University Press, New York, 1993.

5. Juran and Gryna, *Quality Planning and Analysis*, 2nd Ed., McGraw-Hill Book Company, New York, 1980, pg. 85.

6. Beauregard, Mikulak, and Olson. *A Practical Guide to Statistical Quality Improvement: Opening up the Statistical Toolbox*. Van Nostrand Reinhold, New York, 1992, Pg. 312.

7. NSWC/Indian Head Division 3320 Memo, Ser 3320I/062, Subj: Evaluation of EX-99 LOVA Grains by Scanning Electron Microscopy, Indian Head, MD, 16 Mar 2000.

8. NSWC/Indian Head Division 3320 Memo, Ser 3320I/064, Subj: Characterization of EX-99 White Spots, Indian Head, MD, 17 Mar 2000.

This page intentionally left blank.

DISTRIBUTION

COMMANDER NAVAL SEA SYSTEMS COMMAND ATTN TECHNICAL LIBRARY 2531 JEFFERSON DAVIS HIGHWAY ARLINGTON VA 2241-5160	1
COMMANDER US ARMY ARMAMENT RESEARCH DEVELOPMENT AND ENGINEERING CENTER PICATINNY ARSENAL ATTN D. FAIR (BLDG 172) DOVER NJ 07806-5000	1
COMMANDER US ARMY ARMAMENT RESEARCH DEVELOPMENT & ENGINEERING CTR PICATINNY ARSENAL ATTN AMSTA-AR-WEA(V GONSALVES) DOVER NJ 07806-5000	1
UNIVERSITY OF MARYLAND DEPT OF MECHANICAL ENGINEERING ATTN DR. D. BIGIO COLLEGE PARK MD 20742	1
UNIVERSITY OF MARYLAND DEPT OF MECHANICAL ENGINEERING ATTN DR. H. BRUCK COLLEGE PARK MD 20742	1
ADMINISTRATOR DEFENSE TECH INFO CTR ATTN DTIC-OCP 8725 JOHN J KINGMAN RD STE 0944 FT BELVOIR VA 22060-6218	1 CD
JHU/CPIA ATTN SECURITY OFFICER 10630 LITTLE PATUXENT PKWY STE 202 COLUMBIA MD 21044-3200	1 CD

Internal

MT	1
PM2 (A JOHNSON)	2
TD	1
TDE (MITCHELL)	1
041 (DALTON)	1
20	1
210	1
2150P (V PLUMMER)	2
230 (J MCGOVERN)	2
90	1
920	1
9311 (K NEWMAN)	1
940	1
9420 (R NUCKOLS)	1
9420A (C MURPHY)	3
9420B (W NEWTON)	2
9420C (S RICHMAN)	1
9420D (R MUSCATO)	1
9420H (S LENAHAN)	1
9420J (M GALLANT)	5
9420K (R THOMPSON)	1
9420L (S PRICKETT)	1
9420P (W THOMAS)	1
9440C (R CRAMER)	1
9440W (C WALSH)	1
9450J (S JOHNSON)	1

IHDIV Intranet access:

PM	1
PMK (KAVETSKY)	1
PM3 (A DUONG)	1
TI1	1
TI3	1
3320I (R NICOL)	1
90C2 (K BURROUGHS)	1
90R (P CARPENTER)	1
9410 (D CICHRA)	1
9420M (M MICHIENZI)	1
9440 (D ELSTRODT)	1
950 (W REED)	1

This page intentionally left blank.